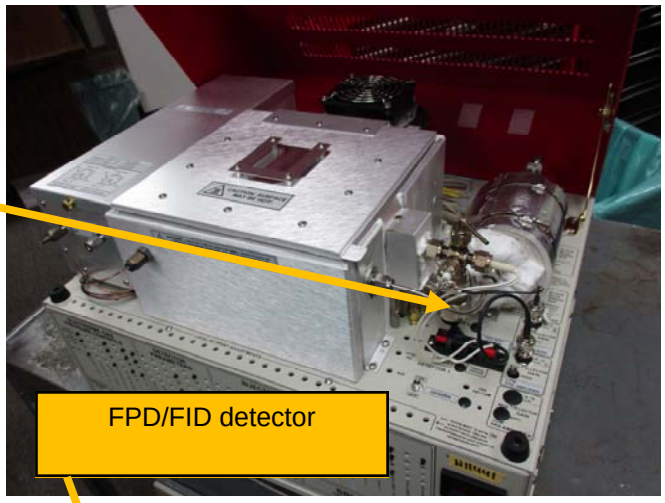
Method 16A GC configuration

March 2019

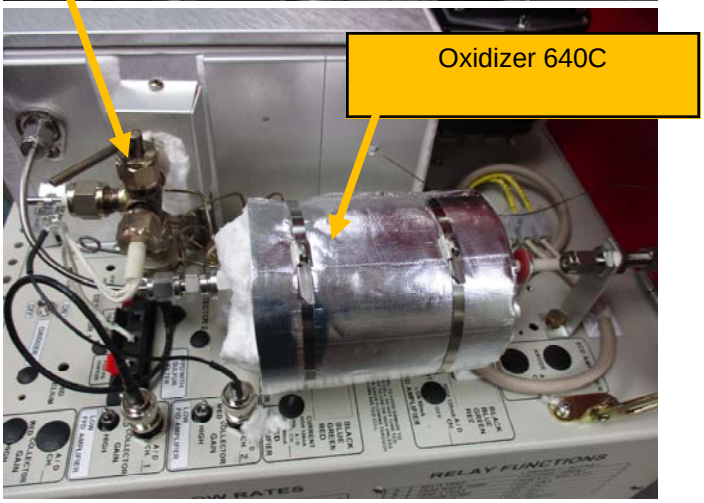
The SRI model 8610C is configured to test for Total Sulfur per EPA Method 16A.

In this method all sulfurs are oxidized to SO2 which is detected by an FPD/FID detector.

The oxidizer furnace is integrated into the GC for easy operation and setup.

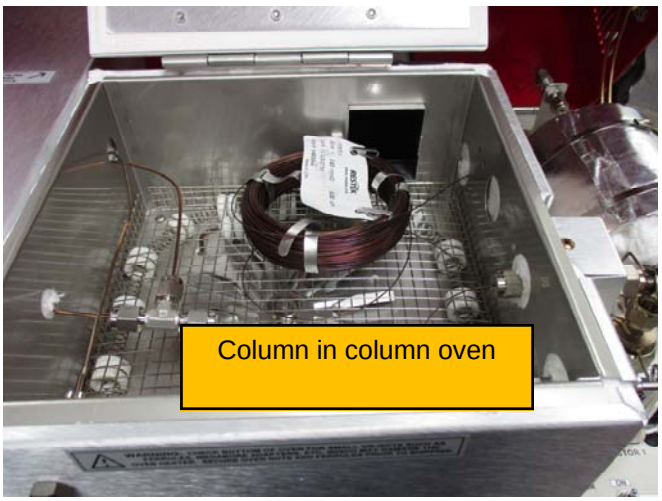


The oxidizer temperature can be set between 0 and 180. Because of the platinum thermoc-couple the actual temperature is 4 times higher than the readout. So a readout temperature of 160 is actually 640C.



Therorectically any column that can separate the CO2 and water away from the SO2 peak can be used. We used a 60 meter MXT1 5 micron film column for this particular system.

This column is very inert and seems to give good results, but it co-clutes COS and SO2. This make it hard to verify that any COS sample has really converted to SO2. Other columns will be evaluated in future systems.



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The sample is delivered to the column by a 10 port gas sampling valve. The sample valve flow joins with a 1ml/minute flow of room air at this "tee" in the column oven.

The air flow can be adjusted with a front panel control (Air 2) which is set up so 10psi=1ml/ minute.

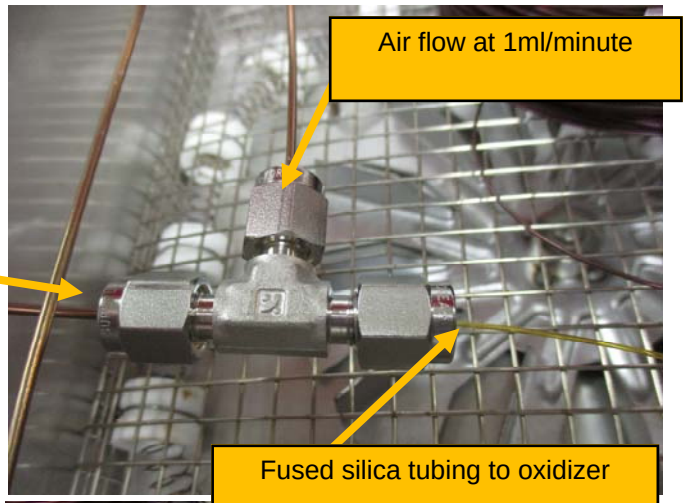
The combined sample and air flow through a length of .53mm id fused silica tubing about 20" (50cm) long. The tubing is fused silica because metal tubing does not seem to work even though it would be more robust if it did work.

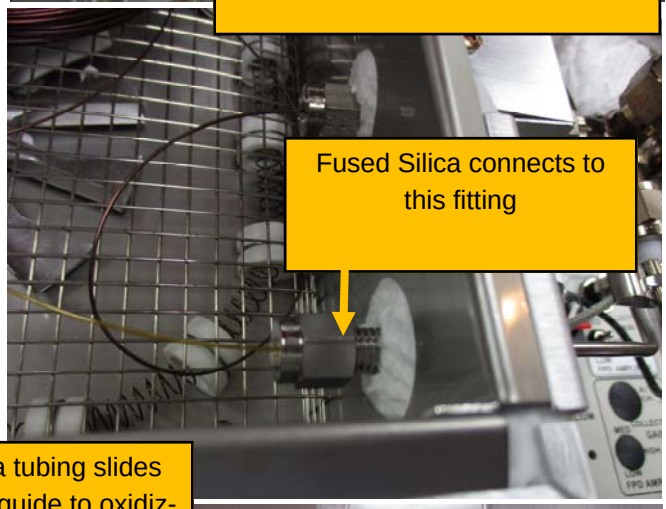
A guide tube is provided to keep the fused silica tubing from breaking where it has to bend to connect to the ixidizer.

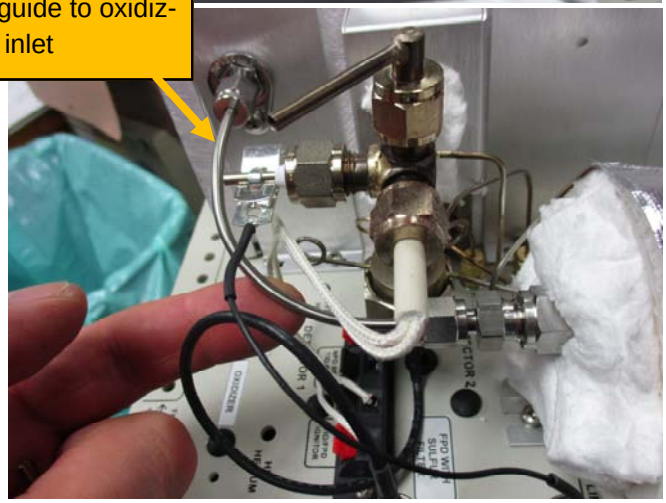
The fused silica tubing slides through the 1/8 inch guideway and enters the oxidizer right in the center of the oxidizer tube entrance. Since the brown polyimide coating on the fused silica will burn off inside the high temperature oxidizer, its important the fused silica tubing be perfectly straight and under zero sideways stress.

The fused silica is secured by soft graphite ferrules. The ferrules just hold the tubing in place.

They don't have to be very tight since there is no gas pressure to contain. Just past finger tight is all that's necessary.







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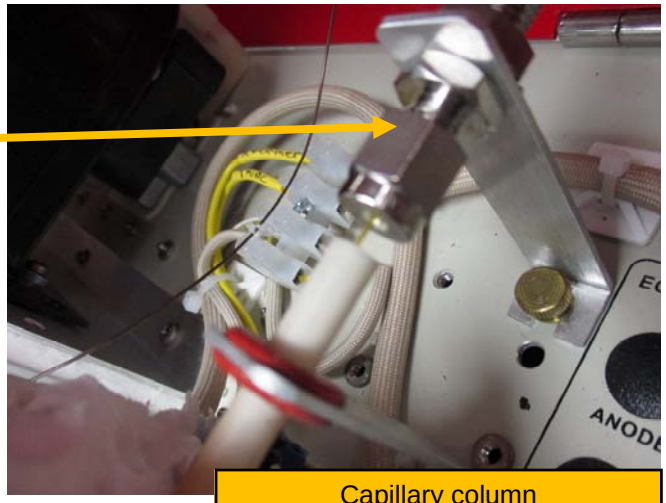
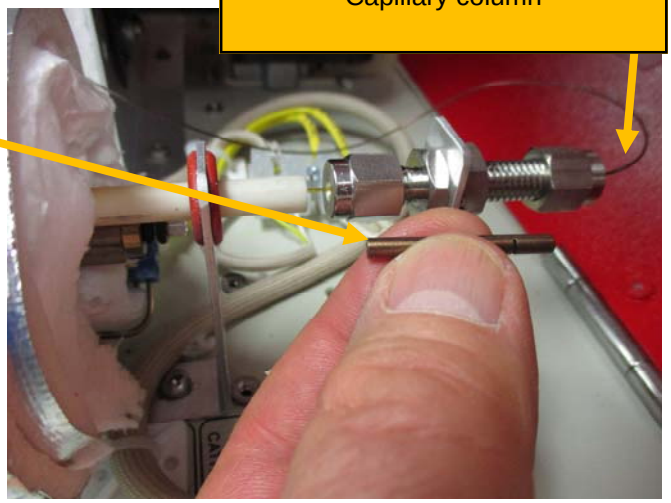
The end of the fused silica tubing exits the oxidizer and terminates in the middle of this 1/8" bulkhead fitting. The bulkhead fitting and the bracket it is mounted on must hold the fused silica tubing right in the middle of the oxidizer so it does not touch the wall of the oxidizer. In this case, the 1/8" to .8mm soft graphite reducing ferrules ferrule must make a seal again the 10-20psi carrier gas pressure so it has to be tightened snugly but not overtightened.



Capillary column

Use the wide bore adapter to connect the 60 meter MXT1 capillary column. The fused silica tubing and capillary column have the same outer diameter (.8mm) so the side bore adapter allows the two tubes to be aligned and touching each other inside the bulkhead fitting. This makes for a low dead volume connection.



The capillary column exits the bulkhead fitting and is routed through the GCs oven wall into the column oven and then to the detector.